# XMaS

the UK-CRG

# BEAMLINE HANDBOOK

## Contents

# 1. Introduction

The beamline uses as its source a dipole bending magnet that delivers photons with critical energy of 10 keV. The beam is delivered to the experimental area through an evacuated system containing: (i) crystal optics for monochromatising, in a tunable way, the x-ray beam emerging from the storage ring; (ii) a mirror for focusing the monochromatic beam onto the sample in the experimental hutch; (iii) several sets of slits for beam defining and scatter attenuation; (iv) safety shutter to enable safe working in the experimental hutch (for sample mounting/alignment etc) while keeping the optics illuminated and stable in the optics hutch.

The main experimental tool available to Users in the experimental hutch is an 11-axes (8-circle) diffractometer. To this can be attached other equipment, such as a cryostat, a magnet, polarisation or conventional analysers, etc. Between the final Be window of the beamline and the diffractometer is a double mirror facility for the attenuation of harmonics and a flexible system for the introduction of beam monitors, anti-scatter slits, attenuators, etc. Most of the path between final Be window and the sample can be evacuated to reduce air scatter.

A more complete description of the beamline is contained from the XMaS web page, accessible through the ESRF web page at:
http://www.esrf.fr/UsersAndScience/Experiments/CRG/BM28/

# 2. Safety

## 2.1 The beamline safety system

### 2.1.1 Introduction

The **front end** (main beam line shutter inside the machine tunnel) can be opened only if the requirements from both the PSS ("Personal Safety System") and the PLC ("Programmable Logic Controller" are fulfilled for the optics hutch.
The **safety shutter** (beam shutter situated between the main optics and the experimental hutch) can be opened only if the experimental hutch has been interlocked (PSS of the experimental hutch).

### 2.1.2 Personal Safety System PSS

The PSS controls the opening and closing of the hutches. No beam shutter can be opened unless the hutch has been properly interlocked. This aspect of the safety is fully covered by the safety training. Any external user coming for experiment on the XMaS beamline MUST follow the **safety training** EACH TIME he comes to the ESRF (waived if he has undergone such training during the previous 3 months at the ESRF), and before he is allowed to perform any work at the beamline. After the safety training, he must check with his local contact on the interlock procedure at XMaS.

### 2.1.3 Programmable Logic Controller PLC

In addition to the PSS, most beamline optical components are protected by the "PLC" which is actually a vacuum interlock system. Valves between components can only be opened if the vacuum pressure on both sides is below $10^{-6}$ mbar. The front end can be opened only if all valves are open.

## 2.2 Hazardous equipment

### 2.2.1 Beryllium

Beryllium in a finely divided state is a serious toxin. Great care should be exercised when it is necessary to manipulate equipment containing beryllium – e.g. cryostat shrouds; detector windows; etc. Section §2.4.3 details the procedure to follow should there occur a breakage of a beryllium component.

### 2.2.2 Radioactive source

Can only be borrowed from the Safety Group by a member of the beamline staff.
A radiation warning sign must be placed on the door of the room in which it is used.
A further sign should be placed in front of the source when in use.
Ask for guidance regarding the use of the source – handling should be done by beamline staff only.

### 2.2.3 Hazardous sample or sample environment

The ESRF's Safety Group read all proposal forms, rank experiments on the basis of safety and issues approval forms coded in colour:

- green for low risk;
- yellow for higher risk
- red for significant risk

Section §3.1.2 below describes the meaning to the user of these approval forms.

### 2.2.4 Liquid nitrogen

When pouring liquid nitrogen from vessels or otherwise manipulating it where risk of personal contact exists safety gloves and safety glasses must be worn. These are provided in the experimental hutch.

### 2.2.5 Crane

This is a manually operated device in the experimental hutch. It has a safe load limit of 500kg. Harness straps are available in the hutch. Always consult the local contact before attempting to manoeuvre very heavy equipment. When not in use the crane tackle should be parked in a corner of the hutch to avoid accidental collisions with personnel.

## 2.3 Sensitive items

### 2.3.1 Diffractometer (collisions!!!)

This is a high precision instrument constructed to meet an exacting specification (see the XMaS web page) and maintain good repeatability and precise coincidence of the centres of the axes. It also has the attribute of great flexibility. **But** this leaves very wide scope for collisions between the several moving elements and thus the possibility of serious damage to the instrument. Great care and vigilance is required of the user – set limits where possible and be alert, especially when operating with two elements in close proximity.

#### 2.3.2 Scintillation counters

Be window!
An electrical potential of 2kV exists when switched on. Do not connect/disconnect before switching off the power supply in the control cabin.
Serious damage can be caused to the scintillation counter if the incident flux is too high. The "oops" command [§3.4] will quickly insert a lead blind into the beam if necessary. This command should also be used when moving between reflections.

#### 2.3.3 Canberra solid state detector

Be window
"oops"
refill twice a day

### 2.4 What to do if….

#### 2.4.1 You cannot interlock the hutch

If you have checked the obvious things - door closed properly, PSS buttons pressed in the correct order - call your local contact or the hall operator (25 25) outside normal working hours.

#### 2.4.2 You have a collision with the diffractometer

Your local contact must be informed, or if unavailable, another member of the beamline staff. Do not attempt continuing your experiment without consultation.

#### 2.4.3 You break a Be shroud or window.

Leave the room immediately. Close the door behind you
Call your local contact - Outside working hours: telephone the hall operator (25-25)

#### 2.4.4 The Canberra solid state detector warms up

Switch off the power supply
Wait – several hours }
Refill } consult a beamline staff member if this seems necessary
Wait – several hours }

#### 2.4.5 spec is stuck

If spec got stuck while executing a command
"^c" aborts the command
"^d" exits spec

### 2.4.6 Need to abort a spec command (mistype, moving too far…)

"^c" aborts the command
"^d" exits spec
Avoid "^\": this "high emergency" key combination may run you into some bigger trouble:
- impossible to restart spec
- data points lost
- motor positions lost

## 2.5 Who can help you

### 2.5.1 Local contact

All experiments have a "local contact" assigned who is normally one of the beamline scientists. This person is your first call for all questions and problems concerning the preparation, setting-up and conduct of the experiment. You should address him to obtain technical support. He will decide how to proceed and will direct your request to the responsible technicians or support groups.
You should also contact him if you get stuck with the experiment. He will assist you solve the problem and possibly contact other persons or groups as appropriate. Normally, he will be available during working hours. He may also be called at home outside working hours, if he has explicitly agreed this.
You can find the office telephone numbers of potential local contacts in the table below.

### 2.5.2 Floor operator

The floor operator serves as a link between the control room and the experimenters at the beamlines. He is also responsible for safety problems, i.e. problems with the front end, the interlock system etc. In general, he is not responsible for technical problems specific to the beamline or to your experiment or for computing problems, but he will assist you in getting support from third persons.
During USM (User Service Mode) the floor operator is available 24 hours a day, including during the weekends, on an 8 hour shift basis. You can call him on the telephone extension 25-25. The call is automatically redirected to a beep; he will call you back within a few minutes.

### 2.5.3 Important Phone Numbers

| Correspondent | Function | ESRF extn. | Home no. | Mobile no. |
|---|---|---|---|---|
| Emergency | General emergency | 10 | | |
| Health Service | Infirmary | 33 | | |
| Floor Operator | (beep) | 2525 | | |
| Control Room | | 2490 | | |
| Laurence Bouchenoire | BL scientist | 2580 | 04 76 87 43 52 | 06 73 03 40 38 |
| Simon Brown | BL scientist | 2197 | 04 76 53 41 77 | 06 72 15 05 48 |
| Danny Mannix | BL Scientist | 2437 | 04 38 12 07 33 | 06 76 86 37 25 |
| David Paul | BL coordinator | 2436 | 04 76 35 46 75 | 06 89 08 30 67 |
| Paul Thompson | BL technical support | 2797 | 04 76 84 57 54 | 06 75 19 94 41 |
| To contact Users | phone by Work Stn. | 2796 | | |

# 3 General Handling of the Beamline

If everything works and you can interlock both the optics and the experimental hutch, you have to open both the front end and the safety shutter to get the x-ray beam to your sample.

## 3.1 Front End

### 3.1.1 "Manual" mode

Opening or closing the front end is simply done by clicking on "Open Front End"' or "Stop Beam" in the ID application normally running on the right hand window of the workstation. However, the opening of the front end will only be accepted if several conditions are fulfilled (indicated by green squares in the appropriate windows), in particular:

- The storage ring must be in the "Beam Delivery Ready" status, which is displayed in the "ID application" window - this is not the case when the electron beam is lost, the orbit is not stable or if there is a beam injection problem.
- The PSS must be enabled (as displayed in the FE values window), i.e. the optics hutch must be interlocked.
- All beamline in-line vacuum valves must be open.

Closing of the shutter can also be done by operation of the button on the rh side of the PSS panel –useful if you arrive at the hutch having forgotten to close it at the workstation.

In the case of beam loss, you will be normally asked to close the front end by a message appearing on the screen. Please follow that instruction – the control room will shut it if you do not. After recovery of the beam you have to reopen it again by clicking on "Open Front End".

Whenever you leave the beamline unattended, you must switch the front end operation mode to "**automatic mode**" beforehand. It is done by clicking on the item **"Set Automatic Mode**" in the "Commands" menu of the ID application. It is recommended that you switch off automatic mode when you are present at the beamline (click on "**Remove Automatic Mode**" in the "**commands**" menu of the ID application.

### 3.1.2 "Automatic mode"

This unattended-operation mode is authorised if you have a green safety approval form (signed at the beginning of the experiment, and posted on the white board opposite the end of the beamline in the circulation freeway). It may also be authorised if a yellow form is issued, but only following the approval of the safety officer having checked your experimental set-up. Unattended mode is not authorised when a red safety approval form is in force. This concerns, for example, experiments with dangerous samples (trans-uranium, virus…) or a dangerous sample environment. In this case, there must be at least 2 persons present at the beamline 24 hours/day (even during PSS tests, injections…).

Where automatic mode is authorised it is possible to operate the beamline without anyone being present at the experiment. However, the following must be observed.

You must inform the floor operator (**25-25**) and fill in one of the small forms, that you will find on the white board - the filled in form is posted into a slot on the white board.

Automatic mode is limited to **72 hours**, after which the front end automatically closes.

If you do leave the beamline, you must either set “automatic mode” or close the front end! Leaving the beamline with the front end open and without setting automatic mode is a violation of ESRF safety regulations – the floor operator will close the front end if he finds the beamline in this state!

### 3.2 Safety shutter

The safety shutter is a part of the PSS and blocks the beam for the whole experimental hutch. You can only enter the experimental hutch if this shutter is closed.
The safety shutter can only be opened if the hutch has been properly interlocked, as demonstrated in the beamline safety training.

Opening and closing the safety shutter is done from within spec using the commands “**shopen**” and “**shclose**”. Closing the shutter can also be done at the PSS panel.

### 3.3 Vacuum

This application displays the status of the different pneumatic valves and vacuum gauges along the beamline. These items are part of the interlock system, as described. It is generally started from the “controls” window, either directly on “xmas3” or on the “xmd28“ terminal. All valves have to be opened (coloured green) before the front end can be opened. To open a valve, the status of the section vacuum must be determined – valves will not open at pressures $>10^{-6}$ mbar (check with your local contact if you are not sure!).

### 3.4 Lead blind

This lead blind is not part of the beamline safety system but is very helpful for the safety of the detectors. Being rather small it has the advantage of reacting much faster than the front end or safety shutters. It is actually one of the attenuators and can be driven into the beam either manually from the electronic rack (switch labelled “lead” on one of the blue electronic cards commanding the attenuators) or from fourc by typing “**oops**” (an alias for “**atten 128**”). If you have set the lead blind manually, you also have to remove it manually before it can be operated from fourc again.

## 4 Computers

### 4.1 Introduction

In the control cabin you will find, facing the experimental hutch, a Sun workstation, “xmas3”. This is the computer devoted to the control of the beamline. The HP workstations, “xmas2” and “xmas1”, in the adjoining cabin and the PCs on the beamline are networked and can be connected to xmas3 via telnet or by using the Exceed application (PCs only).

#### 4.1.1 Getting the Computers Started and Stopped

Normally, all relevant computers will be logged in and running the appropriate programs. However, it cannot be excluded that a program has to be aborted or a computer logged off due to certain failures or breakdowns. Should this occur, the following description will help things get restarted.

NEVER close a window using the file menu or the key combination “Alt F4”: this could well close hidden processes that were started from that window. Use the command “**exit**” instead.

NEVER click on the “exit” button in the bottom panel of the xmas3 workstation. This will start a general logoff and kill all running applications!

#### 4.1.2 Logging into xmas3

| | |
|---|---|
| User: | opd28 (this stands for “Operator D28”) |
| Password: | tonic28 |

Use lower case keys only, UNIX is case sensitive!

### 4.2 Applications for general beamline operation

Once logged into xmas3, several beamline applications can be started by pressing the left mouse button and releasing it on the desired item. They are split under the headings:

| | |
|---|---|
| Machine and Front end: | Id Application |
| | FE diagnostics display on xmas3 |
| | FE diagnostics display on xmd28 |
| Beamline Control | Vacuum |
| | Shutter |
| | Sigmon Gauges |
| | Sigmon generic |
| Spec | Fourc |
| | Mirror |
| | cc5 |
| | tubeslits |

Note: as a matter of convenience only spec applications/windows are normally opened in the LH screen and the beamline control and ID applications are opened in the RH screen. The mouse cursor moves progressively from screen to screen but windows cannot be moved between screens.

### 4.2.1 ID Application

This application controls the opening/closing of the "front end" shutter and displays the status of the front end and also of the storage ring, together with messages from the control room, etc. To start the application, in an uncluttered window on the RH screen, hold down the left mouse button, move to the topic "Id Application" in the "Machine and Front End" menu and then release the mouse button. Windows for the ``FE Status'' may be opened by clicking on the appropriate topics in the drop-down menu "View" of the ID application

Whenever there is a need to terminate the ID application you can do so by clicking on "Exit" in the "File" menu. A complete logoff can be obtained by pressing the middle mouse button and releasing it on the topic "Logout" in the menu showing up.

### 4.2.2 FE diagnostics

### 4.2.3 Vacuum

This application displays the status of the different pneumatic valves and vacuum gauges along the beamline. These items are part of the interlock system, as described in §2.1 above. It is generally started from the "controls" window, either directly on "xmas3" or on the "xmd28" terminal. All valves have to be opened (green logo) before the front end can be opened. To open a valve, you first have to check the status of the vacuum (and check with your local contact if you are not sure!)

### 4.2.4 Shutter

With the middle mouse button you can open general utilities installed on the workstation, in particular terminal windows to launch commands to xmas3. You can close any window on your screen by typing exit.

### 4.2.5 Sigmon Gauges

An application displaying the vacuum gauge readings along the beamline – normally only consulted by users if difficultly is experienced in opening the front end shutter.

### 4.2.6 Spec

"**spec**" is the program package controlling the experiment. Four different versions of spec can be running:

> "**mirror**" dealing with the mirror and the different sets of slits (labelled s1 to s5) along the beamline. In most cases, mirror is only used at the beginning of the experiment, and then **only by the local contact**.
>
> "**fourc**" to actually run the experiment. This is the "four-circle" version of spec, allowing one to work in reciprocal space, using the four-circle geometry and the angles $2\theta$, $\theta$ and $\chi$ as they are defined in Busing & Levy (Acta Cryst 22, 457-464 (1967)).

“**cc5**” for setting up the harmonics-rejection mirrors. Most users will have no need of these and they are normally parked away from the beam. A procedure for using these mirrors is described in section 9.8 below.

“**tubeslits**” for setting the aperture positions and gaps when these are installed, either upstream of the diffractometer or on the 2θ arm of the diffractometer. Section 9.6 describes their use.

Normally there is no need for intervention during the start-up of fourc. However the program might ask you if you want to modify the control register if the stored motor positions are not identical to the currently read-out ones. Actions allowed to Users do **not** involve any modification of the controllers, i.e. answer **no** to the question and **write down the values** - in case of doubt consult your local contact. Otherwise, the fourc prompt appears after some time and possibly some error messages which can be ignored in most cases. From then on the diffractometer can be operated via spec as described in section 5 below.

Normally, all actions in spec can be cancelled pressing the “Ctrl” and “C” keys simultaneously, “^C”. Completely exiting spec in a regular fashion can be done by typing quit.

**Attention**: If “^C” does not work you should try to exit spec by “^D” and restart it. If this does not work either and you really have to stop the ongoing action you should consult your local contact and consider an emergency stop of spec as described in the emergency section §2.4.6 of this handbook.

## 4.3 Data analysis software

### 4.3.1 cplot

For immediately plotting and printing the measured data a plot program called cplot - which is set up to be able to read data files generated by spec - has been installed on xmas3. At the “opd28” prompt you can start cplot by typing cplot, which should give you a “cplot” prompt after some time. From here you can generate plots and send them to the beamline printer following the instructions in section §5.8 To exit from cplot simply type exit, which will ask you for a confirmation first and then terminate your cplotl session if the answer was “yes”.

### 4.3.2 xop

This is a set of routines that can be used to calculate/plot many useful parameters – e.g. reflectivity, absorption, transmission curves for many materials.

### 4.3.3 mathlab

A full version of this widely used software is available on the PC in the cabin containing the beamline workstation.

# 5 fourc

## 5.1 Introduction

The diffractometer is operated by the software system **spec** (from Certified Scientific Software), an extremely powerful, flexible and widely used application that is also easy to learn. Thus it is widely used on beamlines at the ESRF. The following section describes the very basic operations normally needed to run an experiment. For more sophisticated tasks, or for a more comprehensive description, the spec manual can be consulted. This is available on the beamline.

### 5.1.1 Using the Terminals

The Sun terminal, xmas3, provides certain practical features. Most of them work even when spec is running; the essential ones will be explained here:

To see what was displayed previously on your terminal window, you can scroll back by moving the mouse on the left edge of this window with the middle mouse button pressed.

To retrieve previous commands you use the cursor key with the arrow pointing upwards. By subsequently pressing this key all the commands still stored in the buffer will show up one after the other in the current command line.
Furthermore, spec provides some command line editing features using the left and right arrows.

You can also copy text from one part of the screen to another by highlighting the text to be copied with the left mouse button pressed, moving the mouse arrow to the desired destination and then pressing the middle mouse button to release the mouse buffer, i.e. the highlighted text.

The workstation, xmas3 is running the operating system UNIX. If you want to work at UNIX level and you are not familiar with it, you can find documentation at the beamline telling you how to perform the most basic tasks in UNIX, such as changing directories, handling files, etc.

### 5.1.2 Getting help from spec

**If you don't know the acronym of a command you can get general help from spec by typing "help". Help about special features developed at the ESRF is obtained with the command "help local".**

**If you know the command's name but cannot remember the syntax, entering the command without parameters will give you the signification of the different parameters.**

You can print the definition of each command as programmed in spec by the prdef command. For instance, if you want to know more about the dscan command, you can type

```
prdef dscan
```

This will give you the source code of the definition of this command, which might help you to find out what it actually does. If you can't remember the exact name of a command or want to see a list of related commands --- for example all scan commands you can use the command lsdef

lsdef *scan

which will give a list of all commands containing the string “scan”.

### 5.1.3 A few tricks

!! executes the last command again } but no
!string executes the last command starting with “string” } editing

spec also provides the print (“p”) which can evaluate numerical expressions and can therefore be used as a kind of calculator (“p 3*sin(PI/2)” will answer with 0

To escape to UNIX without terminating the current spec session, type “u” - this creates a UNIX subshell. The “exit” command in the UNIX shell will resume the spec session.

“pon” (≡printer on) opens the buffer for the hardcopy. Everything now appearing on the screen is written into this buffer.
“poff” (≡printer off) closes the buffer and starts the printout.

“hi” returns a list of all previously launched commands.
The number of commands to be listed can be specified by a parameter, which avoids “flooding” the window with all previously used commands – typing “hi n”, will list the last n commands.

It is a good idea to make a hardcopy from time to time (using the pon and poff commands) of the information provided by the wh, pa or wa commands. This hardcopy can aid the recovery of a previous situation if you somehow got lost.
If you want to re-run a scan without having to type the parameter list again you can either scroll back the previous commands using the up arrow, or use the following shortcuts:
“!!” will recall the last command, and start it,
“!dscan” will for example recall the last command beginning with the string “dscan”, and execute it.
“help readline” will give you more information on this feature.

You can launch several commands in one line by separating them with semicolons “;”. The commands will be executed sequentially without further intervention. An example would be “ct; mvr tth 2; mvr phi -1; ct” which counts at the present position, moves two motors and then counts again.

“newfile” opens a new data file. This is highly recommended at the startup of the experiment and at least every 200-300 scans to avoid overloading cplot. “newfile” will prompt you for the name of your data file. This name has to be entered with its full directory information. The data files on xmas3 are generally grouped by runs. The data from run 01_3, for example, are grouped in the sub-directory: /data/xmas11/run01_3

Accordingly, all command files (“macros”) from run 01_3 would be stored in the directory: /data/xmas11/usermacros/run01_3.

## 5.2 Motors

Tables 1 - 4 summarise all the motors defined in fourc and their units. The conventions for the positive direction, zero and unit are the following:

All translations are expressed in millimetres.

Most rotations are expressed in degrees. However, some are in milliradians. This is specified in the tables.

The four-circle angles follow the conventions from Busing & Levy (Acta Cryst 22, 457-464 (1967)) – except for the sign of the $\chi$ rotation.

The individual slit blades are described as “front”, “back”, “up” and “down” and are positive on opening, ie moving away from the center.

The slits “pseudo-motors” are described as “vertical gap”, “vertical offset”, “horizontal gap”, “horizontal offset”. Moving a pseudo motor actually moves two blades at the same time. To avoid collision, the backlash is set on opening.

The positive directions are upward and away from the ring.

<table>
<tr><th colspan="4">fourc motors</th></tr>
<tr><td>Mnemonic</td><td>Units</td><td colspan="2">Comments</td></tr>
<tr><td>vth</td><td>deg</td><td colspan="2" rowspan="6">Standard 4-circle motors<br>hth, htth (resp. vth, vtth)<br>are named th, tth in horizontal<br>(resp. vertical) scattering geometry</td></tr>
<tr><td>vtth</td><td>deg</td></tr>
<tr><td>hth</td><td>deg</td></tr>
<tr><td>htth</td><td>deg</td></tr>
<tr><td>chi</td><td>deg</td></tr>
<tr><td>phi</td><td>deg</td></tr>
<tr><td>tta</td><td>deg</td><td colspan="2">Conventional analyser 2θ</td></tr>
<tr><td>tha</td><td>deg</td><td colspan="2">·· ·· θ</td></tr>
<tr><td>mono</td><td>deg</td><td colspan="2">mono θ</td></tr>
<tr><td>hgt</td><td>mm</td><td colspan="2">position of mono 2nd crystal</td></tr>
<tr><td>xtilt</td><td>mrad</td><td colspan="2">mono 2nd crystal – tilt about surface axis in the vertical scattering plane</td></tr>
<tr><td>ytilt</td><td>mrad</td><td colspan="2">·· ·· ·· – ·· ·· ·· ·· ⊥ to the scattering plane</td></tr>
<tr><td>lgm</td><td>mm</td><td colspan="2" rowspan="3">individual actuators for the 2nd crystal</td></tr>
<tr><td>lgf</td><td>mm</td></tr>
<tr><td>lgb</td><td>mm</td></tr>
<tr><td>inout</td><td>mm</td><td colspan="2">diode position (intensity monitor) 83 → in, 10 → out</td></tr>
<tr><td>hsf</td><td>mm</td><td>huber slit front</td><td rowspan="4">positive on opening</td></tr>
<tr><td>hsb</td><td>mm</td><td>·· ·· back</td></tr>
<tr><td>hsu</td><td>mm</td><td>·· ·· up</td></tr>
<tr><td>hsd</td><td>mm</td><td>·· ·· down</td></tr>
<tr><td>hsvg</td><td>mm</td><td colspan="2">vertical gap (= hsu + hsd)</td></tr>
<tr><td>hsvo</td><td>mm</td><td colspan="2">vertical offset</td></tr>
<tr><td>hshg</td><td>mm</td><td colspan="2">horizontal gap</td></tr>
<tr><td>hsho</td><td>mm</td><td colspan="2">horizontal offset</td></tr>
<tr><td colspan="4">the above repeated for the detector slits : dsf;…………dsho</td></tr>
<tr><td>zup</td><td>mm</td><td colspan="2">diffractometer upstream vertical actuator</td></tr>
<tr><td>zdown</td><td>mm</td><td colspan="2">·· downstream ·· ··</td></tr>
<tr><td>xdiff</td><td>mm</td><td colspan="2">diffractometer horizontal translation ⊥ beam</td></tr>
<tr><td>zdiff</td><td>mm</td><td colspan="2">·· vertical ·· ·· ··</td></tr>
<tr><td>thdiff</td><td>mrad</td><td colspan="2">tilt (horizontal axis ⊥ beam)</td></tr>
<tr><td>cyb_win</td><td>units</td><td colspan="2">cyberstar detector SCA window</td></tr>
<tr><td>eta</td><td>deg</td><td colspan="2">polarisation analyser rotation around beam</td></tr>
<tr><td>thp</td><td>deg</td><td colspan="2">·· ·· θ</td></tr>
<tr><td>ttp</td><td>mm</td><td colspan="2">·· ·· 2θ</td></tr>
<tr><td>cryx</td><td>mm</td><td colspan="2">sample/cryostat mount x-motion</td></tr>
<tr><td>cryy</td><td>mm</td><td colspan="2">sample/cryostat mount y-motion</td></tr>
<tr><td>cryz</td><td>mm</td><td colspan="2">sample/cryostat mount z-motion</td></tr>
<tr><td>tsx</td><td>mm</td><td colspan="2">tubeslits assembly horizontal translation</td></tr>
<tr><td>tsz</td><td>mm</td><td colspan="2">tubeslits assembly vertical translation</td></tr>
</table>

**Table 1**

The mnemonic for the motor is a string, at least two characters long, needed to address the motor with commands. The name of the motor is used for the output of FOURC.
For clarity, the motors have been sorted into the following groups:

Monochromator
Diffractometer
Slits

Motor Names at the diffractometer}

| | | |
|---|---|---|
| tth | 2 Theta | 2θ of the four-circle geometry |
| th | Theta | θ of the four-circle geometry |
| chi | Chi | χ of the four-circle geometry |
| phi | Phi | φ of the four-circle geometry |

Table 1 refers to a standard configuration of the diffractometer, but for a particular experiment some motors may not be installed while others may have been added.

| Mirror motors | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Mirror motors – must **NOT** driven by the User unless specified by the local contact | | | | | | | | | |
| mmid | | mfrt | | | mbck | | | xup | |
| xdown | | mxtilt | | | mytilt | | | mhgt | |
| | | | | | | | | | |
| s1 slits | mm | s1f | s1b | s1u | s1d | s1vg | s1vo | s1hg | s1ho |
| s2 .. | .. | s2f | .. | .. | .. | .. | .. | .. | .. |
| .. | .. | .. | .. | .. | .. | .. | .. | .. | .. |
| s4 .. | .. | .. | .. | .. | .. | .. | .. | .. | .. |
| s5 .. | .. | .. | .. | .. | .. | .. | .. | .. | .. |
| same conventions as for the huber slits, hs.. | | | | | | | | | |

**Table 2**

| cc5 | | |
|---|---|---|
| Mnemonic | Units | Comments |
| ppth | deg | phase plate θ rotation |
| ppchi | deg | phase plate chi rotation |
| ppx | mm | phase plate horizontal translation |
| ppz | mm | phase plate vertical translation |
| m1th | mrad | upstream harmonic mirror grazing incidence angle |
| m1tilt | mrad | upstream harmonic mirror lateral tilt |
| hgt1 | mm | upstream harmonic mirror height |
| m2th | mrad | downstream harmonic mirror grazing incidence angle |
| m2tilt | mrad | downstream harmonic mirror lateral tilt |
| hgt2 | mm | downstream harmonic mirror height |
| mtrans | mm | lateral translation for selection of coated/uncoated surfaces |
| mono | | ?? |

**Table 3**

| Tubeslits motors | | |
|---|---|---|
| Mnemonic | Units | Comments |
| tsvg | mm | Vertical gap (= tsu + tsd) |
| tsvo | mm | Vertical offset |
| tshg | mm | Horizontal gap |
| tsho | mm | Horizontal offset |

**Table 4**

## 5.3 Orientation matrix

"setlat"

"or0" the leading direction

"or1" the secondary direction. Spec will adjust positions in this direction to agree with the given lattice constants

"pa" (≡parameters) returns the lattice parameters and the two reflections used to define the orientation matrix

"sa" saves your orientation matrix in a file

A trick: to help you start you can give an approximate orientation matrix in "simulate mode": type "onsim"; calculate angles for simple non-parallel reflections; drive angles (5.5); use or0 and or1; then type "offsim". Note: you can reset any limits you want when in simulate mode – they will be forgotten when returning to operation mode.

## 5.4 Positions

### 5.4.1 Motor position

"wa" (≡where all) returns the current positions of all motors

"wm xxx" (≡where motor xxx – e.g. wm tth) returns the position of the motor with mnemonic xxx (user value, which is the value used by fourc, as well as dial value, which is an absolute physical value) and the soft limits on this motor. "wm" can have more than on parameter, to ask simultaneously for the positions of several motors.

"set motor value" resets the user value of the motor, ie the offset between the user value and the dial value. The soft limits are also modified by the same offset.

"set_dial motor value" resets the dial value. Only the beamline staff are allowed to reset dial values.

"set_lm motor lower_limit upper_limit" resets the soft limits on the user value.

### 5.4.2 Reciprocal space

"wh" (≡where) displays your position in reciprocal space (hkl) as well as the four diffractometer angles (2θ=tth, θ=th' χ,chi, φ=phi)

"ca H K L"

"setmode" }
} see the spec manual

"freeze"          }

### 5.4.3 Energy

"getE" returns the energy in keV. The calculation is based on the value of the d-spacing of the monochromator (contained in the locked variable "g_mo_d") and the rotation angle of the monochromator ("mono").

## 5.5 Move

### 5.5.1 Move a motor

"mv motor position" moves the motor to the desired position (absolute move), provided it is inside the soft limits and no limit switch is activated before the position is reached.

"mvr motor increment" moves the motor by the given increment (relative move), with the same restrictions as for the "mv" command.

"umv" and "umvr" (≡updated move and updated relative move) have the same effect as "mv" and "mvr", but display the motor position on the screen during movement.

"_mv" and "_mvr" also have a similar syntax, but gives the control back before the command is completed. This may be useful if you want to perform some calculations in fourc while the motor is moving.

"tw motor increment" (≡tweak) allows consecutive relative moves by the given increment. + and – change the direction, any other key exits.

"ctw motor increment n" combines the "tw" command with a counting for n seconds.

"an chi_value phi_value" and "uan chi_value phi_value"

"pl chi_value phi_value" and "upl chi_value phi_value"

"m2ax motor1 value1 motor2 value2"

### 5.5.2 Move in reciprocal space

"br H K L" and "ubr H K L"
It is strongly recommended  to calculate (ca H K L) before moving

### 5.5.3 Change energy

"moveE"          Energy is in keV

analyser: "blmenu"

## 5.6 Count

"ct" returns the counting rates measured in all detectors during the preset time (generally one second)

"ct n" returns the counting rates accumulated during n seconds.

"mcaacq" accumulates the out put from the multi-channel analyser (mca). "q" stops it. More details about the use of the solid state detector and the mca will be given in §9.1.

"mcasetup"

"mcaon" }
} switches on/off mca reading
"mcaoff" }

"mcaguion" }
} switches on/off graphical user interface
"mcaguioff" )

## 5.7 Scan

### 5.7.1 Scans output

The format of the scan output is mainly defined by the commands "counters" and "plotselect".

"counters" will ask for the counters chosen as the detector (after the sample) and as the monitor (before the sample). The detector and the monitor will appear in the data file as the last and one-before-last column respectively. They are actually inverted in the screen output.

"plotselect" deals with the screen output during the scan. "plotselect" without parameters will give a list of the available counters, and ask for a choice. "plotselect ccc" will define the counter with mnemonic "ccc" as the one to be plotted.

"splot" and "pplot" are also very useful at this level. They are described in more detail in §5.8.

### 5.7.2 Scan motors

spec supports two kinds of motor scans: absolute scans and relative scans.

"ascan"

"dscan" or "lup" (≡line-up)

"a2scan" and "d2scan" are used to scan two motors simultaneously (absolute and relative scans respectively).

"a3scan" and "d3scan" are the equivalent scans of 3 motors.

"th2th" is a simultaneous relative scan of tth and th (the parameters are the tth range, and the th range will be half the tth range)

"mesh" defines a grid scan. The parameters are absolute values.

#### 5.7.3 Scan in reciprocal space

"hklscan" (If the command is typed without parameters spec displays the syntax)

"hscan", "kscan" and "lscan"

"hklmesh"

#### 5.7.4 Energy scan

"Escan"

"Escan…fixQ"

Analyser (3-axis or polarisation): "blmenu"

#### 5.7.5 Scan analysis

CEN

pl_xMAX

pl_xCOM

### 5.8 Print and plot

"pon" and "poff" used to print output from the screen, e.g. commands in the sequence, "pon" "wa" "poff" will print the table of motor positions.

"plotselect."

"splot" (screenplot) often useful for replotting a scan to the screen following the use of "plotselect" to change the detector output used in the last scan

"pplot" sends the latest scan to the printer

"cpsetup"

### 5.9 Command Files

## 5.10 Emergency

You can abort a scan - as with any other action in spec –with a “^C”. The data points already measured will not be lost as they are already stored in the data file. The aborted scan can be resumed by the command “resume” or “scan_on” (both commands are completely equivalent).

## 5.11 Other features

### 5.11.1 Relative soft limits
### 5.11.2 Safety shutter

## 5.12 Attenuators

“atten”

“oops”

## 5.13 Temperature control

“ontemp”
“te”
“tempmode”
“pid”
“tempgain”
“tempdiff”
“tempint”
“heater”

## 5.14 Magnet

# 6 Setting up

The following is a guide for new users and an aide memoire for experienced users. The order in which the following text sections are written is an indication as to the sequence to follow at the start of an experiment.

## 6.1 Energy selection

It is possible for users to change energy without assistance from the local contact. However, when beam-time follows a period of machine shutdown it is advisable to have the local contact select the initial energy and he will also verify that optimum alignment of the beamline is maintained. Subsequent energy changes can be performed by the user although this procedure is only necessary if the change in energy is significant, e.g. >200eV. The procedure is as follows:

1) Drive the photodiode (pico1) into the beam (mv inout 85).

2) Plot photodiode to screen (plotselect pico1).

3) Scan second crystal relative to first (dscan lgm -0.01 0.01 40 1).

4) Move second crystal to be parallel to first (mv lgm CEN).

5) Rotate both crystals to select * keV photons (moveE *).

6) Adjust the separation of the first and second crystal for constant output offset (move_hgt).

7) Scan second crystal relative to first (dscan lgm -0.01 0.01 40 1).

8) Move second crystal to be parallel to first (mv lgm CEN).

9) Drive the photodiode (pico1) out of the beam (mv inout 10).

10) Plot monitor to screen (plotselect mon).

11) Huber slit-set (hs) to zero vertical and horizontal offset (or to values of previously observed offsets).

12) Transverse tilt scan of second crystal to steer the reflected beam through hs (dscan xtilt -0.4 0.4 40 1).

13) Steer beam through hs (mv xtilt CEN).

14) Drive the photodiode (pico1) into the beam (mv inout 82.5).

15) Plot photodiode to screen (plotselect pico1).

16) Scan second crystal relative to first (dscan lgm -0.01 0.01 40 1).

17) Move second crystal to be parallel to first (mv lgm CEN).

18) Drive the photodiode (pico1) out of the beam (mv inout 10).

**NOTE:** Any change in energy <200ev from one established using the above procedure can be simply accomplished using step (5) above – the crystals retain their alignment and the offset is small enough not to upset the overall beamline alignment.

## 6.2 Conditioning of the incident beam

### 6.2.1 Primary slits

The primary slit-set (s1) select the vertical and horizontal extent of the incident white-beam onto the monochromator. The 'wide open' positions (maximum flux) for the s1 slit-set are s1vg=5, s1hg=70. When the full extent of the beam is not required, the s1 gaps can be reduced. This will decrease and improve the focussed spot by reducing the thermal distortion due to the heat-load on the first crystal of the monochromator.

The synchrotron radiation is linearly polarised on orbit with circularly polarised components above and below orbit. Consequently, one can increase the degree of the linear polarisation by reducing the vertical extent of the beam. This is again best done by reducing s1vg.

#### 6.2.2 Monochromator

Section 6.1 describes the procedure for energy selection.

#### 6.2.3 Mirror

The mirror should never be moved by users. The various movements and tilts for this mirror are adjusted by the local contact in the initial set-up to secure optimum focus at the sample position. The mirror adjustments are completed before final setting of slits s4, hs, ts, ds, etc. Any subsequent "tweaking" of the mirror is very likely to cause significant misalignment of the BL!

#### 6.2.4 Secondary slits

Secondary slit-sets, s2 and s4 may be scanned to reduce background. The slit-set s4 should be positioned to cut out scatter and seems to have the most marked effect on background. Any further scatter produced from the blades of the s4 slit-set may be reduced by suitable positioning (scanning) of the hs slit-set, situated just before the monitor. In addition a removable slit assembly, "tubeslits" can be located downstream of the hs slit set, very close to the sample if required - see section §9.6.

### 6.3 Centring the pin on the diffractometer

The sample goniometer pin is, of necessity, very sharp and care should be exercised in order to avoid both personal injury and damage to the point. Always replace the plastic sheath when it is not in use, a practice that will serve to protect both the user and the point of the pin, which is easily damaged.

Once the pin is mounted in the sample goniometer (or on the displex, as appropriate), set the phi axis vertical and rotate phi to one extreme of its travel. Rotate theta-vertical to position the cryx (or cryy) motion to be in line with the leveller and lock. Adjust the optical leveller crosshairs coincident with the point of the pin. Rotate phi 180 deg. The centre of rotation will be midway between the crosshairs and the new position of the pin. Readjust the crosshairs to the estimated half way position. Move cryy (or cryx) to reposition the pin onto the crosshairs. Return phi to its original position. Should the pin not now be in line with the crosshairs the procedure will need to be repeated until rotating phi by 180 deg leaves the pin aligned with the crosshairs in both positions. Once achieved, rotate phi by 90 deg. and adjust cryx (or cryy) until the point of the pin ia aligned with the crosshairs. The pin is now centred on the phi circle. As a check, rotate phi from one extreme to the other and confirm that the point of the pin appears not to deviate.

Now centre the pin on the chi circle (and thus, effectively, for all the remaining circles of the diffractometer). Rotate chi by 180 deg. And move the cross-hairs on the leveller to the new position of the pin with the vertical translation dial, noting the difference in scale reading between the 2 positions. Thus again, the centre of rotation of chi is the position mid-way between the chi=0 and chi=180 position. Use either the goniometer vertical translation knob or the cryz motion to re-position the pin point coincident with the cross-hairs. Check the pin point remains on the cross hairs when chi is rotated back through 180 deg. and iterate as necessary until it does. Pin alignment is now complete.

### 6.4 Aligning the diffractometer to the beam

Place a piece of green burn paper (stored in the fridge) in front of the pin. Interlock the hutch and expose the paper to the unattenuated beam for about 10 seconds (the one-line command "shopen;ct 10;shclose" will achieve this). Gently press the paper onto the point of the pin to produce an embossed reference to compare with the burn mark. Adjust xdiff (horizontal translation, positive direction is toward the storage ring) and zdiff (vertical translation, positive is up) to bring the pin to the burn position – beware, relative motions, "umvr" are appropriate here! Eventually, final precise alignment will be aided by placing the burn paper behind the pin to produce a shadow on the burn mark – a centered shadow is the goal. Finally, reposition the cross-hairs of the leveller coincident with the pin-point. This becomes your reference for accurate sample positioning – take care not to knock the leveller from now on!

### 6.5 Aligning the sample on the diffractometer

#### 6.5.1 Room temperature

Reflection from the mirror causes the beam to arrive at the diffractometer with a mean 9 mrad slope angle (~ 0.5 deg). Normally, the diffractometer has a 9 mrad compensating tilt applied (pseudo-motor thdiff). It is often useful, when aligning a sample, to temporarily drive th -0.5 deg, for true horizontal positioning of the sample. Mount the sample in the sample goniometer and adjust the translations and arcs such that the sample surface is on the leveller horizontal line for all phi values and the leveller vertical line is at the centre of the sample – for vertical scattering.

#### 6.5.2 low temperature

The sample should be mounted on a 6 mm diameter copper stub which will fit directly into a standard cryostat mount. More information about the sample mounting arrangements are shown here:

http://www.esrf.fr/UsersAndScience/Experiments/CRG/BM28/Sample_environment/

Note: the standard displex's have different mounting threads, for affixing the cryostat mounts, from the 2 low-temperature-adapted displex's.

## 6.6 Finding reflections

### 6.6.1 setlat, primary reflection and or0

In order for fourc to construct an appropriate orientation matrix one must enter the sample crystal lattice parameters a, b, c, alpha, beta and gamma. These are real-space parameters and can be entered with the command setlat, which will prompt the user for the above parameters.

Once the lattice parameters have been entered one may calculate a given reflection with use of the ca macro. For example, ca 0 0 2, for a sample with the c axis in the bisector position should give sensible values of theta and two-theta, ie theta is approximately one half of two-theta. If this is not the case, the command onsim will put fourc into simulation mode. One can then simulate the movement of theta and two-theta (and all other motors) such that one is close to the expected values for a given reflection. Typing or0 0 0 2 in simulation mode will cause fourc to recalculate the orientation matrix such that it corresponds to the known sample orientation (one may also enter the second reflection, or1, in this manner). The command offsim turns off simulation mode but retains the orientation matrix.

To move to the primary reflection one may type, for example, ubr 0 0 2. Small movements of theta, two-theta, chi and phi may be used to 'peak-up' on the reflection. This may be achieved by scanning, but it is generally quicker in the first instance to use the audible feature on the rate meters and move the motors manually until the required reflection is approximately peaked-up. Once the reflection has been optimised, one should enter this as the primary reflection with the command or0 H K L.

### 6.6.2 Off-axis reflection and or1

To complete the orientation matrix, one must enter a second non-collinear reflection, for example, if the primary reflection was the (0 0 2), the any reflection with non zero H or K will suffice. Here again, if the approximate motor positions are known, invoke simulation mode in fourc and enter or1 H K L to allow calculation of actual motor positions. Once the appropriate reflection has been located and peaked-up the command to enter is or1 H K L. One can now ubr to any reflection within the geometrical limitations of the diffractometer.

## 6.7 Different Experimental Set-ups

### 6.7.1 Using the conventional analyser

The conventional analyser is a 2-circle (tha and tta) goniometer that clamps to the dovetail slide of the detector arm of the diffractometer. XMaS possesses 2 crystals, Si & Ge 111, for mounting onto a small Huber goniometer stage on the tha circle. The dovetail on the tta arm of the analyser accommodates a detector, either in-line or orthogonal (scattering from a kapton foil) – the latter mounting is essential for the early stages of analyser alignment to avoid beam damage to the detector. Alignment of the analyser should follow the alignment of the beamline. Primary alignment of the analyser concerns ensuring the tta axis lies in the plane of the crystal face. A quick and simple way to do this is to arrange both detector arms, diffractometer and analyser at their respective "02" (in-line) positions and record the count rate in the detector. Place the analyser crystal onto the tha circle with its face horizontal (a small tha scan through the beam establishes true horizontal) and again record the count rate – the crystal face is on-axis when the count rate is halved. Adjust the appropriate linear adjustment under the crystal mount (manual) until this half count rate is achieved.

### 6.7.2 Using the polarisation analyser

The polarisation analyser is a 3-axis, 2-circle + 1-linear motion (thp, eta and ttp), goniometer that clamps to the dovetail slide of the detector arm of the diffractometer. XMaS possesses a range of crystals, premounted in holders that locate onto a small stage on the thp circle – consult the folder, situated in the control cabin, containing lists of available crystals tabulated with beam energies and appropriate Bragg angles. The detector is normally mounted on a linear stage which allows sufficient adjustment to ensure reception of the beam reflected from the crystal. Alignment of the analyser should follow the general alignment of the beamline. An assembly containing a 45$^o$ copper scattering surface behind a pinhole, accurately located at the centre of the analyser thp circle, mounts in the manner as for the crystal holders. the copper surface scatters the incident beam into the detector above. Alignment is achieved by first scanning the diffractometer vertical 2θ, tth and then horizontal 2θ, htt and in both cases aligning upon and setting zero at the peaks of the scans. The selected crystal then replaces the pinhole/scatterer assembly.

### 6.7.3 Grazing incidence experiments

The XMaS diffractometer is well adapted for grazing incidence experiments. The whole instrument can be tilted to match the gradient of the incoming beam (9 mrad). This means the horizontal θ and 2θ circles, hth a htt, then operate in the plane of the beam.

## 7. At the end of the experiment

### 7.1 General

It is the responsibility of the users to remove their sample(s) from the instrument. For samples mounted on the cryostat the compressor must be turned off and the sample stage allowed to warm up (~ 1 hour) before switching off the vacuum pump and removing the shrouds – thermal shocks can damage the cryostat. Users should tidy-up the hutch (replace tools in the racks provided) and likewise the control cabin. It is expected that users whose experiment ended at 8 am on the final day will have effectively cleared the beamline by mid morning of that day.
The logbook can be photocopied on one of the photocopiers located on the mezzanines above the stairwell that serves the upper level offices – the nearest is close to the end of the beamline across the circulation freeway.
Please remember to fill in and return to a beamline staff member the form for reporting stoppages that occurred during your experiment. The data collected from the completed forms is key to our efforts to provide Users with a trouble-free and improving service.
Do please complete asap an experimental report form, now web based – follow the procedure for ESRF experiments and be sure to correctly fill in the BL and experiment numbers. It is not necessary to delay doing this until you submit new proposals for beamtime.

### 7.2 User data

The data collected during the experiment is stored in the workstation, xmas3, in various directories on the hard drive dependent on the versions of spec that were running during the experiment. Normally the local contact will have created the appropriate data and log files in consultation with the user during the initial experiment setup. A number of options exist at the end of the experiment to enable the user to transfer this data to his home institution. The current recommended method is transfer by ftp. This is now quite straightforward and reliable with no restriction on the size of data files to be transferred. The procedure for doing this is well described in the ESRF ftp manual pages located:

http://www.esrf.fr/Infrastructure/Computing/Networks/InternetAndTheFirewall/UsersManual/FTP/

Another method for data transfer is by copying the data to cd rom. Users may wish to do this simply as a backup to the ftp method or possibly instead of it. The data and log files should be transferred (by ftp) from xmas3 to the PC, xmas8, located in the same control cabin. Xmas8 contains a cd writer and balnk cds are available on the beamline.

# 8. Print and Plot

## 8.1 cplot

### 8.1.1 Introduction

Data can be plotted with the program, cplot, installed on xmas3. cplot is set up to easily read data files from SPEC. It is an extremely versatile and powerful program system for the production of scientific graphics. However, only the very basic features needed to produce a plain x-y plot will be described in this manual. If you intend to do fancier things, such as some preliminary data treatment or use of smaller plot windows as insets, you should consult the original cplot manual, available at the beamline. The cplot functions and capabilities are summarised in the pages “c-plot command reference” and “c-plot format reference” at the beginning of the cplot manual.

- Help
  - Cplot manual
  - On-line help
- Start cplot
- Read data
- Plot data
- Fit data
- Print
- Command files

## 8.2 specplot

## 8.3 mathlab

This is an application available on the PC in the control cabin. It has an on-line help facility and a full manual is available in the cabin.

# 9. Equipment

## 9.1 Detectors

### 9.1.1 Bicron – SCA

### 9.1.2 Cyberstar

### 9.1.3 Canberra solid state detector

A guide to the startup and use of the mca will be available in due course. A useful guide is available however, on the ESRF's web pages at: http://www.esrf.fr/computing/bliss/gui/mcatcl/mcagui.html

### 9.1.4 Ion chamber

### 9.1.5 Mar detector

### 9.1.6 Peltier-cooled solid state detector

### 9.1.7 Magic eye

A detector with video output showing an image of the x-ray beam on a video screen (shared with the tripod/leveller camera outputs and located in the control cabin). Most used in the focal position on the diffractometer for initial setup by the local contact, to optimise beam focus, and users will not normally have access to it. **Important:** judicial use of the attenuator foils, and additional aluminium sheets if appropriate, will serve to preserve the life of this detector and also enable optimum "glare-free" viewing of the beam.

## 9.2 Magnet and power supply (1 Tesla)

- The magnet can be mounted to produce horizontal (in line or orthogonal to the beam) or vertical magnetic fields.

△ Ensure that the magnet power supply is turned off and unplugged!!!

- Connect the power supply's cables to the magnet
- Connect the water pipes to the magnet
- Turn the water valves ON
- Reconnect the supply cable to the wall socket

**magneton** (turns the magnet power supply on and call the magloop macro)
**kemsetv** "type in the requested value" (the value of the voltage goes from 0 to 1023)
**kemsetc** "type in the requested value" (the value of the current goes from 0 to 700)

△700 is the MAXIMUM value of kemsetc that should be used. Above 700, the magnet could be seriously damaged.

**kemsetup**

- Serial device name (d28/s1/14)? ↵ (the value in brackets indicate the current value)
- Voltage (200)? Type the new value or press ↵ to keep the current value
- Current (0)? Type the new value or press ↵ to keep the current value
- Step (1023)? Type the new value or press ↵ to keep the current value

- Sleep time after step (sec) (0)? Type the new value or press ↵ to keep the current value
- Sleep time after flip (sec) (0.2)? Type the new value or press ↵ to keep the current value

**magsetup** (to enter name of the counters and to define what A, B and R are etc…)

1- Counter setup
   11- Detector……………………..……:<name> - Monitor: <name>
   12- A = det+/mon+………………….. :<magA> - A error: <magAE>
   13- B = det-/mon-………………….…..:<magB> - B error: <magBE >
   14- D=A-B……………….…………….:<magD> - D error: <magDE >
   15- S=A+B……………….…………….:<magS> - S error: <magSE >
   16- R = D/S…………………………….:<magR> - R error: <magRE >
   17- Other counters saved as cnt+, cnt-…:<name> <name>, etc…

2- Acquisition setup
   21- Counting time……………………………. : type the value (in seconds)
   22- Number of cycles …….: type the value
   23- Magnet sequence…………………: e.g. 600 –600 –600 +600 (→ 4 polarities)

3- Save all data to additional file……………...… : <yes>
   31- Filename……………………………. : type the value (in seconds)
   32- Last scan number………………………… : type the new value or press ↵
   33- Save MCA data………………………….. : <yes or no>

4- Magloop on all scans………………………….. : <yes or no>

5- Simulation mode……………………………… : <no>

6- Exit

Option (0)? enter the number corresponding to the parameter you want to change press or ↵ to quit magsetup

NB:

- the word “magloop” is written at the start of each scan if the magloop macro is running. If not, go to magsetup and type <yes> for item 4 (Magloop on all scans)
- spec records the data in three different data files when magloop is running:

1) in the current fourc file: e.g. /data/xmas11/run03_3/experiment.01
   This file records all the average magA, magB, magR, etc… and their corresponding errors. These are the data plotted in the fourc window. This file can also be plotted with newplot.
2) in *.mag file specified in item 31 (Filename)
   e.g. /data/xmas11/run03_3/experiment.01.mag
   This file records each individual magloop. It will contain magA, magB, etc… and also the corresponding error bars for each cycle. This file can be also plotted with newplot.
3) in *.raw file saved at the same place as the *.mag file:
   e.g. . /data/xmas11/run03_3/experiment.01.raw

This file records all the raw data, i.e. for every single polarity.

*Example:*

MAG_CTTIME = 1 (equivalent to going to item 21 in magsetup and typing 1)
MAG_CYCLES = 6 (equivalent to going to item 22 in magsetup and typing 6)
MAG_SEQ = "500 -500" (equivalent to going to item 23 in magsetup and typing 500 -500), (→ 2 polarities)
lscan 0.1 3.7 35 1 (implies that there will be 36 magloop scans)

→ the average magA, magB, magR, etc… and their errors will be saved for each 36 points of the lscan in /data/xmas11/run03_3/experiment.01. (36 ligns in total)

→ each individual magloop scan will be saved in /data/xmas11/run03_3/experiment.01.mag. Each magloop scan will contain magA and magB etc… and their corresponding errors for each cycle. Each scan will thus have 6 ligns.

→ all the raw data (mon, cyber, det, pico3) will be saved for each magloop and each sequence in /data/xmas11/run03_3/experiment.01.raw. Each magloop scan will thus contain 12 ligns (6 cycles x 2 flips)

**magnetoff** (turns the magnet power supply off)

**Problems** when using the magnet:

- if the magnet power supply displays no signs of life, press the green button located on top of the casing.
- check that it is not in "local" - if it is, switch to "remote".
- if you're measuring zero effect from field flipping, check with the Gaussmeter that the magnet is actually reversing the magnetic field. If it is not, call your local contact for assistance.

## 9.3 Attenuators

These are a set of 7 aluminium foils of different thickness and one lead screen. The foils are inserted into the beam with the command "atten xxx" where xxx is any number between 1 and 128 – representing a "logical" sum with resultant foil thickness. The command "atten" displays the current setting of the foil array, as below.

| # | F1 | F2 | F3 | F4 | F5 | F6 | F7 | F8 |
|---|---|---|---|---|---|---|---|---|
| # | 1 | 2 | 4 | 8 | 16 | 32 | 64 | 128 |
| # | 6 | 12.5 | 25 | 50 | 100 | 200 | 400 | Pb |
| 50 | OUT | IN | OUT | OUT | IN | IN | OUT | OUT |

Thus the command "atten 50" has resulted in the insertion of foils of thickness: 12.5, 100 and 200µm. "atten 127" would insert all foils (793.5µm) and "atten 128" inserts the Pb screen (≡ "oops"). "atten 0" removes all foils and screen.

## 9.4 Low temperature cryostat

Two versions of this adaptation of an APD displex exist. One of these will achieve T<2K at all orientations while the other, an earlier prototype, will achieve T<2K only at angles<45° from vertical – reducing to 6K at horizontal and more extreme angles.
Consult appendix A for full diagram-aided instructions on how to install and setup this cryostat.

### 9.5 High temperature cryostat

This displex cryostat features a higher power heating stage enabling an operating temperature range of 10-800K. It operates in a very similar fashion to the standard APD displex.

### 9.6 Tubeslits

These are a 4-blade slit assembly providing a 4x4 mm maximum adjustable aperture that can be placed very close to the sample, either upstream or downstream of it. There is a separate spec application, "tubeslits" for operating this assembly. The blades/aperture are controlled from spec in a similar way to all other slit assemblies. Thus tsvo, tsvg, tsho and tshg refer to the vertical offset and gap, and horizontal offset and gap, respectively – the individual blades can be controlled if thought necessary – see §5.2 for mnemonics. Downstream the assembly becomes the first element on the rail of the diffractometer detector arm. Upstream it locates upon a rail mounted on a motorised x-z mount – motors, tsx and tsz. This enables easy alignment of the small-aperture assembly.
**Note 1:** tsx and tsz operate only from the "fourc" application.
**Note 2:** the mounting rails in the downstream and upstream positions are orthogonal and it should be remembered that the vertical and horizontal senses are correct only for the detector arm mounting. In the upstream position vertical becomes horizontal and vice versa – tsvo, tsvg and tsx are axes in the same sense while tsho, tshg and tsz are axes in the same sense!

### 9.7 Motorised sample mount

Primarily designed for manipulation of the cryostat to better enable the maintenance of sample alignment over the cooling cycle, this mounting is routinely used for all sample environments. The motor names are cryx, cryy and cryz and are controlled from the "fourc" spec application. All motions are effected through cams and are thus oscillatory in nature – this can result in confusion near the end of a cam stroke! The maximum strokes are 3 mm for cryx and cryy  and 5 mm for cryz – no hard limits are provided or are necessary for these motions.
**Note:** The motor cluster on this mount prevents angular movements of phi greater than 183 degrees. This should be borne in mind by users when positioning samples. **Important:** the hard limits must be correctly set for phi to avoid damage to both the mount and the phi and chi circles.

### 9.8 Harmonic rejection mirrors

This is an assembly containing 2 flat mirrors in an evacuable vessel upstream of the huber slits, monitor and attenuators. By positioning these mirrors, to reflect the x-ray beam at the critical angle for the incident beam energy, the higher order components, $\lambda/3$ for example, become severely attenuated –  a factor of $10^{-6}$ being possible. The mirrors have been coated with Rhodium along one (long) half of their reflecting surfaces to enable their use over a wide energy range – coated surfaces for high energy, >9 keV, and uncoated (pyrex) for energies < 9keV.
The following procedure assumes all basic alignment steps for the beamline have been completed, including alignment of the slits, s1 – s4.
Note: it is important to ensure that the detector(s) are correctly windowed on the fundamental!

Using the appropriate chart select both the mirror angle of incidence, a, and the surface material, Rhodium or pyrex, according to the beam energy.

Set s4vg < 0.35a (this is to prevent overfilling the mirror apertures)

move mtrans according to the surface selected: -10 for Rh
10 for pyrex

Line up the huber slits around the beam and move hsvg to 0.2

"umv hgt1 0" (places m1 mirror surface nominally in the beam)

1) move hgt1 in small steps until approx. half cutting the beam (½ counts)

"dscan m1th –1 1 40 1" ) rock m1 to locate the
"umv m1th pl_xMAX" ) surface parallel to the beam

"ascan mtrans –12 12 30 1" ) scan mtrans, adjust m1tilt and
"umvr m1tilt x" ) iterate until scans are level

"umvr hgt1 –1" (remove m1 from the beam)
"dscan hgt1 0 2 40 1" (scan m1 through the beam)
umv hgt1 to the half-cut position

"dscan m1th –1 1 40 1" ) rock the mirror to find
"umv m1th pl_xMAX" ) $\theta = 0$ relative to beam

"set m1u 0"
"set m1m 0"
13) "set m1d 0"

"umv hgt –1" (remove m1 from beam)
"umv hgt2 0" (places m2 mirror surface nominally in the beam)
repeat steps (1) to (13) for mirror 2 – hgt2, m2th, m2tilt, m2u, m2m, m2d.
"umv hgt1 0"
"umv m1th a" (where "a" is the selected incidence angle)
"umv m2th a"
"umv hgt2 0.72*a" (offset m2 to receive reflected beam from m1)
"umvr hsvo –0.72*a" (offset huber slits to receive offset beam from mirrors)
"dscan hsvo –1 1 40 1"
"umv hsvo CEN"
"dscan s4vg –1 1 40 1" (optimise s4 vertical gap for max flux with least harmonic bleed-through)

If the diffractometer had been aligned w.r.t. the beam before inserting the mirrors it will need realignment w.r.t. the offset beam (0.72a).

The harmonic mirrors are removed from the beam with the command,
"mirout"